IS 5182-8 (1976): Methods for measurement of air pollution, Part 8: Sulphation rate [CHD 32: Environmental Protection and Waste Management]

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Indian Standard

METHODS FOR MEASUREMENT OF AIR POLLUTION

PART VIII SULPHATION RATE

(Third Reprint SEPTEMBER 1991)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN. 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3 *April* 1977

AMENDMENT NO. 1 OCTOBER 2000
TO
IS 5182 (PART 8) : 1976 METHODS FOR MEASUREMENT OF AIR POLLUTION

PARTS SULPHATION RATE

(*Page* 3, *clause* **1.1**) — Substitute the following for the existing clause

'1.1 this standard (Part 8) prescribes the methods of measurement of sulphation rate in air, using lead peroxide as one of reagents. Lead peroxide, due to its oxidizing power, converts other compounds such as mercaptans and hydrogen sulphides into sulphates. It also fixes sulphur trioxide and sulfuric acid mist present in the atmosphere. It converts oxides of nitrogen into nitrates.'

(CHD 32)

Reprography Unit, BIS, New Delhi, India

Indian Standard

METHODS FOR MEASUREMENT OF AIR POLLUTION

PART VIII SULPHATION RATE

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Indian Standard

METHODS FOR MEASUREMENT OF AIR POLLUTION

PART VIII SULPHATION RATE

0. FOREWORD

0.1 This Indian Standard (Part VIII) was adopted by the Indian Standards Institution on 27 December 1976, after the draft finalized by the Air Pollution Sectional Committee had been approved by the Chemical Division Council.

0.2 The sulphation measurement technique is a simple and inexpensive method used to determine the average sulphur dioxide concentration for long periods over a number of areas in the same region. It requires minimum of equipment, normally available in all analytical laboratories.

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part VIII) prescribes the methods of measurement of sulphation rate in air.

2. CANDLE PREPARATION

2.1 Reagents

2.1.1 *Lead Peroxide* — analytical reagent grade, powdered.

2.1.2 *Gum Tragacanth* — powdered.

2.1.3 *Ethyl Alcohol* — 95 percent (*v/v*).

2.1.4 *Hydrochloric Acid, Concentrated* — relative density 1.19.

2.1.5 *Sodium Carbonate* (Na_2CO_3) — anhydrous powder.

2.1.6 *Hydrochloric Acid* — 0.05 N.

*Rules for rounding off numerical values (*revised*).

2.1.7 *Barium Chloride Solution* — Dissolve 50 g of barium chloride ($BaCl_3.2H_2O$) in water and dilute to 1 000 ml.

2.2 Procedare

2.2.1 Cut a fabric such as tapestry cloth or stockinette or gauze bandage, according to the size so as to wind the 100 cm^2 area of the surface of a cylindrical plastic or glass tube candle. Wind the fabric around the candle and fix it with a cotton thread; the fabric acts as a support to the paste-coating over the surface of the candle.

2.2.2 Prepare gum tragacanth mucilage by dispersing 5 g of gum tragacanth in 10 ml of alcohol and then add at one time with stirring, 190 ml of water.

2.2.3 Mix 8 g of lead peroxide with 6 ml of gum mucilage to obtain a mixture that is required for a single candle. Apply the entire mix immediately and evenly on the fabric of the candle with the help of a spatula. Care shall be taken to apply the paste only over 100 cm^2 area and not more.

2.2.4 Take another lot of 8 g lead peroxide for the second candle and prepare the second one as in **2.2.3.** Thus follow the procedure separately for each candle.

2.2.5 Place these candles in a bell jar or a large desiccator having a suitable holder for the tubes and containing a small amount of calcium chloride, until the tubes are fairly dry. Do not dry excessively.

3. SAMPLING

3.1 Transfer candles in a carrying case on the first day of the month to observation site. Install the candle in louvered box for exposure for 30 days. On the first day of the next month remove the candles, replace the new ones for another month. Bring back the exposed candles to the laboratory for analysis.

3.2 Treat a control or a blank candle in exactly the similar way as the other cylinders, except that it is stored in a desierator free from atmospheric contamination to make sure that none of the material used in the candle preparation has been contaminated by sulphur dioxide or other sulphurous gases.

4. ANALYSIS OF THE EXPOSED CANDLE

4.1 Gravimetric Method

4.1.1 *Procedure* — Strip off the coated fabric from the cylinder and treat with 5 g of anhydrous sodium carbonate in 100 ml of water, contained in a 400 ml beaker. Allow to stand with occasional stirring for at least 3 hours.

4.1.1.1 Heat the contents nearly to boiling for half an hour, keeping the volume nearly constant by the addition of water (control the flame and keep a close watch on heating operating, avoiding spurting which may result in spoiling the sample).

4.1.1.2 Filter the contents through filter paper Whatman No. 41 or equivalent into another beaker with subsequent 4 to 5 washings with hot water. Refilter, if necessary, to get a clear solution.

4.1.1.3 Neutralize the filtrate with concentrated hydrochloric acid by cautiously adding the acid in small amounts and stirring the solution with a glass rod to prevent frothing. Neutralize to a *p*H range of 3 to 4.

4.1.1.4 Boil the solution for some time and make the volume to about 250 ml. Preferably, two blanks should be prepared from unexposed candles in this way. This solution will contain sulphate in the form of sodium sulphate (Na_2SO_4).

4.1.1.5 Adjust the acidity of the solution with dilute hydrochloric acid to *p*H 4.5 to 5 using *p*H meter. Then add additional 1.5 ml hydrochloric acid and heat the solution to boiling.

4.1.1.6 Remove the burner and while stirring gently, add warm barium chloride solution slowly until precipitation appears to be complete, and leave it overnight. About 10 ml of solution is required for complete precipitation.

4.1.1.7 Filter out the barium sulphate precipitate in a washed and ignited Gooch crucible and wash the precipitate with small portions of warm distilled water until the washings are free from chloride as indicated by testing with silver nitrate reagent.

4.1.1.8 Dry the crucible with the residue and ignite at 800°C for 1 hour. Cool in a desiccator and weigh.

4.1.2 *Calculation*

$$\text{Sulphation rate (mg of } SO_3 \text{)/100 cm}^2\text{/day} = \frac{M \times 343}{D}$$

where —

M = mass in grams of residue, and

D = number of days of exposure.

4.2 Turbidimetric Method

4.2.1 *Principle* — Sulphate ion is precipitated in hydrochloric acid medium with barium chloride so as to form barium sulphate, the absorbance of which is proportional to the concentration of the sulphate present.

42.2 *Apparatus*

4.2.2.1 *Spectrophoiometer*

4.2.2.2 *Stop-watch*

4.2.2.3 *Measuring spoon* — spatula of 0.6 ml capacity.

4.2.3 *Reagents*

4.2.3.1 *Conditioning medium* — Dissolve 75 g of sodium chloride in 300 ml of distilled water. To this add 30 ml of concentrated hydrochloric acid and 100 ml of 95 percent ethyl or *iso*propyl alcohol. Mix and to this mixture add 50 ml of glycerol.

4.2.3.2 *Barium chloride crystals* — 20 to 30 mesh size.

4.2.3.3 *Standard sulphate solution* — Dissolve 0.147 9 g of anhydrous sodium sulphate in water and dilute to 1 000 ml in volumetric flask. One millilitre of this solution corresponds to 0.10 mg of sulphate (SO_4).

4.2.4 *Calibration*

4.2.4.1 Take ten (or more) 100-ml beakers in a series, and number them appropriately. Add the following measured quantities of standard sulphate solution:

Beaker No.	1	2	3	4	5	6	7	8	9	10
Volume in ml of standard sulphate solution	0	1	2.5	5	10	15	20	25	30	35

4.2.4.2 To each beaker add measured quantity of distilled water so as to make the total volume of the solution to 50 ml.

Beaker No. (*Containing* SO_4)	*Volume in ml of Water to be Added to it*
1	50
2	49
3	47 5
4	45
5	40
6	35
7	30
8	25
9	20
10	15

4.2.4.3 Thus, we get the following quantities in mg of SO_4 in 50 ml:

Beaker No.	1	2	3	4	5	6	7	8	9	10
Quantity of SO_4 in mg/50 ml distilled water	0	0.1	0 25	0.5	1.0	1.5	2.0	2.5	3.0	3.5

4.2.4.4 Add 50 ml of conditioning medium to each of the beakers and mix. Add a spatula full of barium chloride crystals to the first beaker and immediately swirl (clockwise) the contents of the beaker by hand with uniform speed for exactly 1 minute. Keep the beaker on table for 3 minutes to acquire maximum absorbance. Adjust the spectrophotometer to 100 percent transmission with distilled water using 420 mμ wave length.

4.2.4.5 After 3 minutes, but before 10 minutes after thorough mixing, tranafer the contents of the beaker to the cuvette and read its percent transmission.

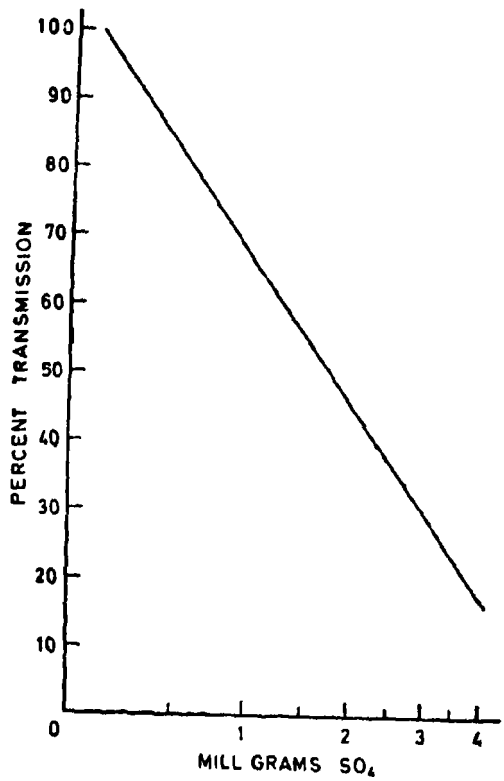
4.2.4.6 Repeat the procedure for the other beakers in turn and record the readings in the following manner:

Beaker Mo.	mg.SO_4 *in 50 ml*	*Percent Transmission*
Blank	Nil	100
1	0	99 to 100
2	0.1	98
3	0.25	93
4	0.50	88
5	1.0	71 to 70
6	1.5	58 to 57
7	2.0	45 to 46
8	— 2.5	36 to 35
9	3.0	29
10	3.5	23 to 22

NOTE — This chart shall be taken as an illustration. The percent transmission may vary slightly in plactice

4.2.4.7 Plot the graph using semilog graph paper.

NOTE — Do not try to locate the point corresponding to first beaker (0 mg.SO_4 50 ml) on the graph. The straight line may not pass through 100 percent transmission. The first and the last few points may not exacily fall on the stranght line.



NOTE 1 — This graph should be taken as illustration.

NOTE 2 — The percent readings may slightly vary in practice.

FIG. 1 CALIBRATION GRAPH FOR SULPHATE DETERMINATION

4.2.5 *Procedure* — Proceed as in **4.1** up to **4.1.1.3.** Boil and cool and make up the volume to 250 ml in a volumetric flask. Preferably two blanks shall be prepared from the exposed candles. It is expected that the blanks and the samples should have the following final volumes at this stage:

Blank 1 and 2	100 ml each
Samples	250 ml

4.2.5.1 For analysis take (in 150-ml beakers) 50 ml of the above solutions or select the aliquot volume and make it up to 50 ml with water.

4.2.5.2 Select such an aliquot that after analysis its percent transmission reading will fall in the range of 90 to 40. This can be achieved by trial and error. Take different aliquots and dilute to 50 ml. Select the aliquot which gives percent transmission reading between 90 and 40.

For test solution the aliquot should be so chosen that this solution should contain SO_4 not exceeding 3 mg SO_4/50 ml of solution.

Generally for blanks, take 50 ml out of 100 ml of the solution directly in the beaker for analysis, and for samples start with 10 ml aliquot out of 250 ml and add 40 ml water to make up the volume to 50 ml.

4.2.5.3 To it add 5 ml of conditioning medium and mix well. Add a spatula full of barium chloride crystals and swirl the solution immediately by hand with uniform rotary motion for 1 minute. Keep the beaker on the table and wait for 3 minutes. In the meantime, adjust the spectrophotometer to 100 percent transmission using 1-cm cuvettes and 420 mμ wave length.

4.2.5.4 Pour the portion of the sample from the beaker into matched cuvette. Measure the transmission on the spectrophotometer. Time factor is very important to observe the reading on the colorimeter (within about 8 minutes from the time of adding barium chloride crystals).

4.2.5.5 Record the transmission reading of blank candle solution as well as that of sample solution by referring them to the prepared calibration curve and find out the corresponding sulphate content in the aliquot taken for analysis.

4.2.6 *Calculations*

4.2.6.1 Find the sulphate content of the blank and the sample aliquots used by referring their transmittance readings to the calibration curve and calculate total sulphate expressed as barium sulphate in milligrams.

$$\text{Sulphation rate, milligram } SO_3 \text{ per } 100 \text{ cm}^2 \text{ per day} = \frac{\text{Total barium sulphate in mg}}{\text{Number of days for which the candle was exposed}} \times 0{\cdot}343$$

4.2.6.2 Sulphation rate record may be maintained as given in Table 1.

4.3 Golorimetric Titration Method

4.3.0 This method may be used as alternate to turbidimetric method (**4.2**).

TABLE 1 FORM FOR SULPHATION RATE RECORD

(*Clause* 4.2.6.2)

SL No.		BLANK 1 AND 2		SAMPLE
1.	Name or the Station			
2.	Date of candle installed			
3.	Date of candle removed			
4.	No. of days of exposure (E)			
5.	Date of analysis			
6.	Total volume of Na_2SO_4 solution, ml	100	100	
7.	Volume of aliquot in ml taken	50	50	
8.	Aliquot factor	2	2	
9.	Percent transmission reading			
10.	a) mg SO_4 in aliquot (L)	L_1	L_2	
	b) Avg. mg SO_4, in aliquot of blank $L_b = \frac{L_1 + L_2}{2}$			
11.	Total mg SO_4, in blank or sample	M_b=		M_s =
12.	Total $BaSO_4$, in mg in blank or sample	N_b =		N_s =
13.	Total $BaSO_4$, in mg of exposed candle due to lulphation			N_2 -N_b
14.	Sulphation rate in mg SO_3/ 100 cm^2/day = $\frac{(N_s - N_b) \times 0{\cdot}343}{\text{Number of days candle exposed}}$			

4.3.1 *Principle* — This method is intended for the analysis of absorption solutions with traces of sulphur. As low as 20 μg of sulphur in the form of sulphate can be determined by this method. The aqueous solution containing 20 to 600 μg of sulphur (as SO_4) is made up to 100 ml with *iso*propyl alcohol and titrated photometrically with standard barium perchlorate solution using methylene blue-thorin mixed indicator against identical reagent blank containing 20 ml of distilled water in place of the sample. The end-point of the titration is found graphically by taking it as the point of intersection of two straight branches of the curve.

4.3.2 *Apparatus*

4.3.2.1 *Colorimeter* — suitable one with two green filters (VG 9) and two sample cells.

4.3.2.2 *Micro-burette* — of 10 ml capacity.

4.3.2.3 ***p***H *meter*

4.3.3 *Reagents*

4.3.3.1 *Barium perchlorate stock solution* — 0 01 N. Dissolve 2.0 g of barium perchlorate [$Ba(ClO_4)_2.3H_2O$] in 200 ml of water and make up to 1 000 ml with *iso*propyl alcohol (*see* IS : 2631-1964*). Adjust the *p*H to 3.5 with 1 : 1 perchloric acid [60 to 70 percent (*m/v*)] using a *p*H meter.

4.3.3.2 *Standard barium perchlorate solution* — 0.004 N. Make up 200 ml of solution prepared in **4.3.3.1** to 500 ml with 80 percent (*v/v*) *iso*propyl alcohol. Standardize this against 5.0 ml of standard 0.005 N sodium sulphate solution. One millilitre of this solution contains 64 μg of sulphur.

4.3.3.3 *Hydrochloric acid* — 0.1 N.

4.3.3.4 *Mixed indicator* — Mix 60 ml of 0.20 percent (*m/v*) thorin aqueous with 40 ml of 0.01 percent (*m/v*) methylene blue aqueous.

4.3.3.5 *Standard sulphate solution* — Weigh 0.355 2 g of dried anhydrous sodium sulphate, dissolve in water and make up to 1 litre. Mix well. One millilitre contains 240 μg of sulphur (as SO_4).

4.3.4 *Procedure*

4.3.4.1 *Standardization of barium perchlorate solution* — Pipette 50 ml of standard sulphate solution in a sample cell. Add 15 ml of water, 80 ml of isopropyl alcohol, 2.0 ml of mixed indicator and 3.5 ml of 0.1 N hydrochloric acid. Mix well with a glass rod and place this cell in right hand compartment of the colorimeter.

4.3.4.2 Prepare a titration blank in another sample cell exactly as in **4.3.4 1** but adding 5 ml of water instead of standard sodium sulphate solution. Mix well with a glass rod and place this cell in left hand compartment of the colorimeter—

4.3.4.3 Switch on the instrument and adjust the setting so that the pointer of the galvanometer reads 50 percent transmittance (*T*).

4.3.4.4 Titrate contents of right hand cell with standard barium perchlorate solution with equal increments of 0.2 ml. After each addition stir the solution and note the percentage transmittance. Titrate until slope of the line drawn after the end-point is reached can be accurately established.

*Specification for *iso*propyl alcohol.

4.3.4.5 Plot percentage transmittance against ml of 0.004 N barium perchlorate added. End point is given by the intercept of the line drawn after the end-point with horizontal base line of titration.

4.3.4.6 Calculate the strength of barium perchlorate as follows:

$$\text{Strength of Ba } (ClO_4)_2 \text{ solution} = \frac{240 \times 5{\cdot}0}{V} \text{ microgram } SO_4/\text{ml}$$

where

V = the volume of litre in ml obtained graphically.

4.3.4.7 *Determination* — Proceed as given in **4.1** to **4.1.1.2**. Neutralize the filtrate with concentrated hydrochloric acid by cautiously adding the acid in small amounts and stirring the solution with a glass rod to prevent frothing. Neutralize to a *p*H range of 5 to 6.

4.3.4.8 Take 20 ml of the solution or select an aliquot volume and make up to 20 ml with water in a sample cell. Add 80 ml of *iso*propyl alcohol, 2 ml of mixed indicator and 3'5 ml of hydrochloric acid and proceed as given in **4.3.4.1** to **4.3.4.5.** Analyse sample blanks similarly.

4.3.4.9 *Calculation* — Calculate total sulphate (as SO_4) by the following:

$$\text{Total sulphate (as } SO_4), \text{ microgram} = \frac{V \times F \times 250}{A}$$

where

V = titre value in ml,

F = equivalence of Ba ($ClO_4)_2$ in microgram SO_4/ml, and

A = aliquot taken in ml.

4.3.4.10 Calculate sulphation rate using the following equation:

$$S_R = \frac{(S - B) \times 0\,833}{1\,000 \times D}$$

where

S_R = sulphation rate in mg SO_3 per 100 cm^2 per day,

S = total sulphate in micrograms SO_4 present in sample,

B = total sulphate in micrograms SO_4 present in blank, and

D = number of days for which the candle was exposed.

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephones: 331 01 31, 331 13 75 Telegrams: Manaksanstha
(Common to all Offices)

Regional Offices:	*Telephone*
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